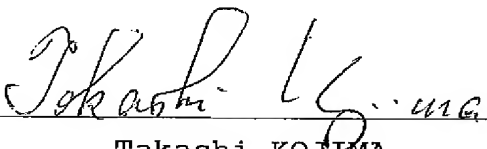


C E R T I F I C A T I O N

I, Takashi KOJIMA of Ginza Ohtsuka Bldg., 2F, 16-12, Ginza 2-chome, Chuo-ku, Tokyo, Japan, hereby certify that I am the translator of the accompanying certified official copy of the documents in respect of an application for a patent filed in Japan on the 20th of November, 2002 and of the official certificate attached thereto, and certify that the following is a true and correct translation to the best of my knowledge and belief.

Dated this 26th day of January, 2007

  
Takashi KOJIMA

(Translation)

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This is to certify that the annexed is a true copy of the following application as filed with this Office.

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[Document]	Specification	1
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2002-336769

[SPECIFICATION]

[TITLE OF THE INVENTION] Heat Resistant Coated Member,  
Making Method, and  
5 Treatment Using the Same

[CLAIMS]

[Claim 1] A heat resistant coated member comprising a  
substrate made of a material selected from the group  
consisting of Mo, Ta, W, Zr, and carbon and a coating of  
10 rare earth-containing oxide thereon, the rare  
earth-containing oxide coating including a surface layer  
having a hardness of at least 50 HV in Vickers hardness.

[Claim 2] The heat resistant coated member of claim 1  
wherein the rare earth-containing oxide coating has a  
15 surface roughness of up to 20  $\mu\text{m}$  in centerline average  
roughness Ra.

[Claim 3] A method for preparing a heat resistant coated  
member comprising  
coating a substrate made of a material selected from  
20 the group consisting of Mo, Ta, W, Zr, and carbon with a  
rare earth-containing oxide, and

heat treating the surface of the coating so that the  
surface has a hardness of at least 50 HV in Vickers  
hardness.

25 [Claim 4] The method for preparing a heat resistant coated  
member of claim 3 wherein the heat treatment is carried out  
at 1,200 to 2,000°C.

[Claim 5] A method of heat treating a powder metallurgical  
metal, cermet or ceramic material, comprising the steps of  
30 placing the material on the heat resistant coated member of  
claim 1 and heat treating the material thereon.

[DETAILED EXPLANATION OF THE INVENTION]

[0001]

[Technical Field of the Invention]

35 This invention relates to a heat resistant coated  
member which is used especially in the sintering or heat  
treatment of metal or ceramic materials in vacuum or an

inert or reducing atmosphere; a method for preparing the same; and a method for the heat treatment of metal or ceramic materials using the coated member.

[0002]

5 [Prior Art]

Powder metallurgy products are generally manufactured by mixing a primary alloy with a binder phase-forming powder, then kneading the mixture, followed by compaction, sintering and post-treatment. The sintering step is carried  
10 out in a vacuum or an inert gas atmosphere, and at an elevated temperature of 1,000 to 1,600°C.

[0003]

In a typical cemented carbide manufacturing process, solid solutions of tungsten carbide with cobalt, titanium  
15 carbide, and tantalum carbide are comminuted and mixed, then subjected to drying and granulation to produce a granulated powder. The powder is then pressed, following which such steps as dewaxing, pre-sintering, sintering and machining are carried out to give the final cemented carbide product.

20 [0004]

Sintering is carried out at or above the temperature at which the cemented carbide liquid phase appears. For example, the eutectic temperature for a ternary WC-Co system is 1,298°C. The sintering temperature is generally within a  
25 range of 1,350 to 1,550°C. In the sintering step, it is important to control the atmosphere so that cemented carbide correctly containing the target amount of carbon may be stably sintered.

[0005]

30 When cemented carbide is produced by sintering at about 1,500°C, green specimens placed on a carbon tray often react with the tray. That is, a process known as carburizing occurs, in which carbon from the tray impregnates the specimen, lowering the strength of the  
35 specimen. A number of attempts have been made to avoid this type of problem, either by choosing another type of tray material or by providing on the surface of the tray a

barrier layer composed of a material that does not react with the green specimen. For example, ceramic powders such as zirconia, alumina and yttria are commonly used when sintering cemented carbide materials. One way of forming a barrier is to scatter the ceramic powder over the tray and use it as a placing powder. Another way is to mix the ceramic powder with a solvent and spray-coat the mixture onto the tray or apply it thereto as a highly viscous slurry. Yet another way is to form a coat by using a thermal spraying or other suitable process to deposit a dense ceramic film onto the tray. Providing such an oxide layer as a barrier layer on the surface of the tray has sometimes helped to prevent reaction of the tray with the specimen.

[0006]

When a graphite tray is used in the sintering of materials to produce cemented carbides or cermets, the graphite tray is coated with a cover layer made of  $Y_2O_3$  containing up to 20% by weight of  $ZrO_2$  or an equivalent volume of another heat resistant oxide such as  $Al_2O_3$  or a combination thereof, and having an average thickness of at least 10  $\mu m$ . (Refer to Patent Document 1.)

[0007]

[Patent Document 1]

JP-A 2000-509102

[0008]

[Problem to be Solved by the Invention]

However, even when a barrier layer is formed on a carbon tray, reaction can occur between the barrier layer and the tray. After one or a few sintering cycles, the barrier layer cracks, fragments and spalls off. Peeling of the coating allows for reaction between the carbon tray and a specimen. During the sintering step, the coating can peel and fragment into pieces which are often introduced into the specimen. Then a fresh coated tray must be used.

[0009]

For the above-described reason, there is a need for a tray having a long lifetime in that when used in sintering, the barrier layer does not react with a specimen or with the tray substrate or peel off, and when used in the sintering of powder metallurgical products, the barrier layer does not react with specimens or peel from the tray substrate even after repeated use.

[0010]

The present invention meets the above need. An object of the present invention is to provide a coated member which exhibits excellent heat resistance, corrosion resistance, and non-reactivity when used especially in the sintering or heat treatment of metal or ceramic materials in vacuum or an inert or reducing atmosphere, to provide a method for preparing the coated member, and to provide a method of heat treatment using the coated member.

[0011]

[Means for Solving the Problem and Embodiment of the Invention]

The present inventors have earnestly studied in order to attain the above object. As a result, it has been found that a heat resistant coated member in which a substrate of a material selected from among Mo, Ta, W, Zr, and carbon is coated with a rare earth-containing oxide exhibits excellent heat resistance, corrosion resistance, and non-reactivity when used in the sintering or heat treatment of a metal or ceramic material in vacuum or an inert or reducing atmosphere. When a surface layer of the rare earth-containing oxide coating has a hardness of at least 50 HV in Vickers hardness, the separation of the oxide coating from the substrate is prohibited. When the surface layer has a surface roughness of up to 20  $\mu\text{m}$  in centerline average roughness  $R_a$ , the coated member is more effective for preventing a ceramic product from deformation during sintering or heat treatment thereon. The present invention has been thus completed.



[0012]

Accordingly, the present invention provides:

(1) A heat resistant coated member comprising a substrate made of a material selected from the group consisting of Mo, Ta, W, Zr, and carbon and a coating of rare earth-containing oxide thereon, the rare earth-containing oxide coating including a surface layer having a hardness of at least 50 HV in Vickers hardness,

(2) A method for preparing a heat resistant coated member comprising

coating a substrate made of a material selected from the group consisting of Mo, Ta, W, Zr, and carbon with a rare earth-containing oxide, and

heat treating the surface of the coating so that the surface has a hardness of at least 50 HV in Vickers hardness, and

(3) A method of heat treating a powder metallurgical metal, cermet or ceramic material, comprising the steps of placing the material on the heat resistant coated member of (1) and heat treating the material thereon.

[0013]

The following is the detailed description of the invention.

The heat resistant coated member of the invention includes a substrate made of a material selected from among molybdenum Mo, tantalum Ta, tungsten W, zirconium Zr, and carbon C and a layer of rare earth-containing oxide coated thereon. The coated member is intended for use especially in the sintering or heat treatment of metals or ceramics in vacuum or an inert or reducing atmosphere to form a product. It is recommended that the type of substrate, the type of coating oxide, and the combination thereof be varied and optimized in accordance with the product itself and the temperature and gas used in sintering and heat treatment.

[0014]

The coated member of the invention is particularly effective as crucibles for melting metal or as jigs for

fabricating and sintering various types of complex oxides. Examples of such jigs include setters, saggers, trays and molds.

[0015]

5 In the invention, the substrate for forming such heat-resistant, corrosion-resistant members used in the sintering or heat treatment of metals and ceramics is made of a material selected from among molybdenum, tantalum, tungsten, zirconium, and carbon.

10 [0016]

When carbon is used as the substrate, the carbon substrate has a density of preferably at least  $1.5 \text{ g/cm}^3$ , more preferably at least  $1.6 \text{ g/cm}^3$ , and most preferably at least  $1.7 \text{ g/cm}^3$ . Note that carbon has a true density of  $2.26 \text{ g/cm}^3$ . At a substrate density of less than  $1.5 \text{ g/cm}^3$ , although the low density provides the substrate with good resistance to thermal shock, the porosity is high, which makes the substrate more likely to adsorb air-borne moisture and carbon dioxide and sometimes results in the release of adsorbed moisture and carbon dioxide in a vacuum.

20 [0017]

When a transparent ceramic such as YAG is sintered, treatment within a temperature range of  $1,500$  to  $1,800^\circ\text{C}$  in a vacuum, an inert atmosphere or a weakly reducing atmosphere tends to give rise to reactions between the substrate material and the coating oxide and to reactions between the coating oxide and the product on account of the elevated temperature. It is therefore important to select a substrate and coating oxide combination that discourages such reactions from arising. At temperatures above  $1,500^\circ\text{C}$  in particular, when carbon is used in the substrate, aluminum and rare-earth elements tend to form carbides in a vacuum or a reducing atmosphere. Under such conditions, it is desirable to use a coated jig in which a molybdenum, tantalum or tungsten substrate is combined with a rare-earth-containing oxide as the oxide coating.

[0018]

The coated members of the present invention have a layer of rare earth-containing oxide coated on the substrate.

5       The rare earth-containing oxide used herein is an oxide containing a rare earth element or elements; that is, an element selected from among those having the atomic numbers 57 to 71.

[0019]

10       Among, the rare earth elements, the substrate is preferably coated with an oxide of at least one rare earth element selected from among Sm, Eu, Gd, Dy, Ho, Er, Tm, Yb, and Lu, more preferably an oxide of Er, Tm, Yb or Lu.

[0020]

15       An oxide of a metal selected from Group 3A to Group 8 elements may be mixed with the rare earth oxide in an amount of up to 20% by weight. More preferably, an oxide of at least one metal selected from among Al, Si, Zr, Fe, Ti, Mn, V and Y is used.

20       [0021]

The oxide used herein is preferably in the form of particles having an average particle size of 10 to 70  $\mu\text{m}$ . The coated member is prepared by plasma spraying or flame spraying a rare earth-containing material in an inert  
25 atmosphere such as argon to deposit a coating of rare earth-containing oxide on the substrate. If necessary, the substrate is surface treated by a suitable technique such as blasting prior to the thermal spraying.

[0022]

30       Alternatively, the coated member is prepared by pressing rare earth-containing oxide particles having an average particle size of 10 to 70  $\mu\text{m}$  in a mold to form a preform, heat treating the preform and attaching it to the substrate.

35       [0023]

The coating of rare earth-containing oxide has a thickness of 0.02 mm to 0.4 mm, more preferably 0.1 mm to

0.2 mm when it is thermally sprayed. At less than 0.02 mm, there is a possibility that on repeated use of the coated member, the substrate may react with the material being sintered. On the other hand, at more than 0.4 mm, thermal shock within the coated oxide film may cause the oxide to delaminate, possibly resulting in contamination of the product. In case the coated member has the heat treated preform attached to the substrate, the thickness of the oxide layer is not particularly limited though a thickness of 0.3 to 10 mm, especially 1 to 5 mm is preferred.

[0024]

In the present invention, the surface of the oxide coating is preferably heat treated in an oxidizing atmosphere, vacuum or inert gas atmosphere at a high temperature of 1,200 to 2,000°C. For example, the surface of the thermally sprayed coating is roasted by an argon/hydrogen plasma flame, whereby the surface of the coating can be smoothed to a surface roughness of 10  $\mu$ m or less.

[0025]

With heat treatment below 1,200°C or without heat treatment, the coating surface may not be smoothed to a desired level of surface roughness. Heat treatment above 2,000°C is undesirable because the oxide coating can be melted or evaporated.

[0026]

Through the heat treatment, the rare earth-containing oxide coating layer in the form of a preform or thermally sprayed coating is increased in hardness, thereby preventing a product being fired from fusing thereto or preventing the coating from peeling off.

[0027]

In the coated member of the present invention, the rare earth-containing oxide coating includes a surface layer having a hardness of at least 50 in Vickers hardness (HV). Preferably the surface layer has a Vickers hardness of at least 80, more preferably at least 100, even more preferably

at least 150. The upper limit of Vickers hardness is not critical, but is generally up to 3000, preferably up to 2500, more preferably up to 2000, even more preferably up to 1500. With too low a surface hardness, when a material on  
5 the coated member is fired, the material being fired fuses to the rare earth-containing oxide coating so that a surface portion of the rare earth-containing oxide coating can eventually be stripped or torn off. With too high a surface hardness, the rare earth-containing oxide coating layer may  
10 crack.

[0028]

Preferably, the surface layer of the oxide coating has a surface roughness of up to 20  $\mu\text{m}$  in centerline average roughness Ra. In the case of a thermally sprayed coating, a  
15 surface roughness (Ra) in the range of 2 to 20  $\mu\text{m}$ , especially in the range of 3 to 10  $\mu\text{m}$  is preferred for effective sintering of a material thereon. At a surface roughness of less than 2  $\mu\text{m}$ , the coating layer is so flat that this may interfere with sintering shrinkage by the material resting  
20 thereon. A surface roughness of more than 20  $\mu\text{m}$  may allow the material to deform during the sintering.

[0029]

When the preform of rare earth-containing oxide particles is heat treated and attached to the substrate to  
25 construct the coated member, the heat treated preform has a very high hardness which permits a powder metallurgical metal, cermet or ceramic material to be effectively sintered on the coated member independent of its surface roughness.

It is also possible that an oxide be thermally sprayed  
30 to form an oxide coating having a surface roughness (Ra) of at least 2  $\mu\text{m}$ , which is optionally surface worked as by polishing.

[0030]

The coated member of the invention is used in the heat  
35 treatment (especially firing or sintering) of metals or ceramics as mentioned above. A metal or ceramic material to

be heat treated is placed on the coated member, whereupon the material is heated or sintered at a temperature of up to 1,800°C, especially 900 to 1,700°C, for 1 to 50 hours. The heat treating or sintering atmosphere is preferably a vacuum or an inert atmosphere having an oxygen partial pressure of not more than 0.01 MPa or a reducing atmosphere.

[0031]

Exemplary metals and ceramics include chromium alloys, molybdenum alloys, tungsten carbide, silicon carbide, silicon nitride, titanium boride, silicon oxide, rare earth-aluminum complex oxides, rare earth-transition metal alloys, titanium alloys, rare earth oxides, and rare earth complex oxides. The coated members of the invention, typically in the form of jigs, are effective especially in the production of tungsten carbide, rare earth oxides, rare earth-aluminum complex oxides, and rare earth-transition metal alloys. More specifically, the coated members of the invention are effective in the production of optically permeable ceramics such as YAG and cemented carbides such as tungsten carbide, the production of Sm-Co alloys, Nd-Fe-B alloys and Sm-Fe-N alloys used in sintered magnets, and the production of Tb-Dy-Fe alloys used in sintered magnetostrictive materials and Er-Ni alloys used in sintered regenerators.

[0032]

Examples of suitable inert atmospheres include argon and nitrogen ( $N_2$ ) atmospheres. Examples of suitable reducing atmospheres include inert gas atmospheres in which a carbon heater is used, and inert gas atmospheres containing also several percent of hydrogen gas. An oxygen partial pressure of not more than 0.01 MPa ensures that the coated members are kept resistant to corrosion during the heat treating or sintering operation.

[0033]

[Effect of the Invention]

In addition to having a good heat resistance, the coated member of the invention also has a good corrosion

resistance and non-reactivity, and can therefore be effectively used for sintering or heat-treating metals or ceramics in a vacuum, an inert atmosphere or a reducing atmosphere. Where the surface layer of the rare  
5 earth-containing oxide coating has a Vickers hardness of at least 50 HV, the rare earth-containing oxide coating is prevented from peeling from the substrate. Where the oxide coating has a surface roughness of up to 20  $\mu\text{m}$  in centerline average roughness  $R_a$ , it becomes effective for preventing a  
10 metal or ceramic product from deforming during sintering or heat treatment.

[0034]

[EXAMPLE]

The following Examples and Comparative Examples are  
15 provided to illustrate the invention, and are not intended to limit the scope thereof.

[0035]

[Examples and Comparative Examples]

Carbon substrates having dimensions of 50×50×5 mm were  
20 furnished. In Examples 1 to 6, the surface of the substrate was roughened by blasting, following which rare earth-containing oxide particles having the compositions and average particle sizes indicated in Table 1 were plasma-sprayed in argon/hydrogen onto the substrate surface,  
25 thereby coating the substrate with a layer of rare earth-containing oxide to form a coated member. Then the sprayed samples were heat treated in vacuum or in argon or roasted by an argon/hydrogen plasma flame, as indicated in Table 2.

30 [0036]

In Examples 7 to 11, an oxide powder whose composition was shown in Table 1 was used and pressed into a preform having dimensions of 60×60×2-5 mm by a die pressing technique. The preform was then heat treated in an  
35 oxidizing atmosphere at 1700°C for 2 hours, obtaining a plate of rare earth oxide. The plate was attached to the

substrate to produce a rare earth oxide-covered member. In Comparative Examples 1 and 2, coated members were similarly produced under the conditions shown in Tables 1 and 2.

[0037]

5       The physical properties of the coated members were measured. The results are shown in Table 1. The compositions were measured using inductively coupled plasma spectroscopy (Seiko SPS-4000). The average particle sizes were measured by a laser diffraction method (Nikkiso FRA).  
10   The physical properties of the thermally sprayed coatings and heat treated preforms were also measured, with the results given below in Table 2. The thickness of the thermally sprayed coating was determined from a cross-sectional image of the coating taken with an optical  
15   microscope. The surface roughness Ra was measured with a surface roughness gauge (SE3500K; Kosaka Laboratory, Ltd.) in accordance with JIS B0601. The Vickers hardness was measured with a digital micro-hardness meter (Matsuzawa SMT-7) in accordance with JIS R1610, after the surface was  
20   mirror finished.

[0038]

      Next, a tungsten carbide powder was mixed with 10 wt% of a cobalt powder and the mixture was pressed into a compact having dimensions of 10×40×3 mm. The compact was  
25   rested on the rare earth oxide-coated member (jig) and sintered in a low vacuum at 1,400°C for 2 hours. The sintering were conducted in a carbon heater furnace in such a pattern that the temperature was ramped up to 1,400°C at a rate of 300°C/h, held at that temperature for a  
30   predetermined length of time, then lowered at a rate of 400°C/h. This sintering cycle was repeated twice, after which the coated member was examined for peeling of the rare earth oxide coating from the substrate, seizure of the coated member to the sample being sintered, and warpage of  
35   the sample. The results are shown in Table 3.



[0039]

Table 1

	Composition (weight ratio)	Average particle size ( $\mu\text{m}$ )	Substrate material	Substrate density ( $\text{g}/\text{cm}^3$ )
Examples 1-3	$\text{Yb}_2\text{O}_3$	40	C	1.7
Examples 4-6	$\text{Er}_2\text{O}_3$	50	C	1.7
Example 7	$\text{Yb}_2\text{O}_3$	40	C	1.7
Example 8	$\text{Dy}_2\text{O}_3$	50	C	1.7
Example 9	$\text{Sm}_2\text{O}_3$	40	C	1.7
Example 10	$\text{Gd}_2\text{O}_3$	40	C	1.7
Example 11	$\text{Gd}_2\text{O}_3 + \text{Al}_2\text{O}_3$ (50:50)	40	C	1.7
Comparative Example 1	$\text{Al}_2\text{O}_3$	40	C	1.7
Comparative Example 2	$\text{Y}_2\text{O}_3$	60	C	1.7

[0040]

Table 2

	Coating layer	Coating thickness (mm)	Heat treating conditions	Before heat treatment		After heat treatment	
				Roughness Ra ( $\mu\text{m}$ )	Hardness (HV)	Roughness Ra ( $\mu\text{m}$ )	Hardness (HV)
Example 1	Yb <sub>2</sub> O <sub>3</sub> sprayed	0.20	no	7	80	7	80
Example 2	Yb <sub>2</sub> O <sub>3</sub> sprayed	0.15	1500° C in vacuum			5	100
Example 3	Yb <sub>2</sub> O <sub>3</sub> sprayed	0.30	plasma flame in air			2	200
Example 4	Er <sub>2</sub> O <sub>3</sub> sprayed	0.15	no	8	65	8	65
Example 5	Er <sub>2</sub> O <sub>3</sub> sprayed	0.20	1600° C in Ar			6	85
Example 6	Er <sub>2</sub> O <sub>3</sub> sprayed	0.20	plasma flame in air			3	160
Example 7	Yb <sub>2</sub> O <sub>3</sub> preform	5	1700° C in air	3	45	0.5	1015
Example 8	Dy <sub>2</sub> O <sub>3</sub> preform	3	1700° C in air	4	40	0.3	650
Example 9	Sm <sub>2</sub> O <sub>3</sub> preform	2	1700° C in air	6	38	1	205
Example 10	Gd <sub>2</sub> O <sub>3</sub> preform	4	1700° C in air	7	48	1.5	310
Example 11	Gd <sub>2</sub> O <sub>3</sub> +Al <sub>2</sub> O <sub>3</sub> preform	5	1700° C in air	5	35	0.8	2130
Comparative Example 1	Al <sub>2</sub> O <sub>3</sub> paste coated	0.2	no	25	30	25	30
Comparative Example 2	Y <sub>2</sub> O <sub>3</sub> preform	3	no	5	40	5	40

[0041]

Table 3

	Coating layer appearance	Seizure of sample	Warpage of sample
Example 1	no peeling	no	0.2 mm
Example 2	no peeling	no	0.1 mm
Example 3	no peeling	no	0.1 mm
Example 4	no peeling	no	0.3 mm
Example 5	no peeling	no	0.2 mm
Example 6	no peeling	no	0.1 mm
Example 7	no peeling	no	0.1 mm
Example 8	no peeling	no	0.1 mm
Example 9	no peeling	no	0.1 mm
Example 10	no peeling	no	0.1 mm
Example 11	no peeling	no	0.2 mm
Comparative Example 1	peeled	seized	1 mm
Comparative Example 2	crazed	no	0.5 mm

5

[0042]

The jigs of Examples 1 to 11 remained unchanged after heat treatment in a carbon heater furnace relative to before treatment. On sintering, the samples did not seize to the jigs and deformed little. By contrast, following heat treatment in a carbon heater furnace, the jigs of Comparative Examples 1 and 2 underwent surface crazing or oxide delamination, leading to corrosion. In Comparative Example 1, the sample seized to the jig and deformed noticeably.

10

15

2002-336769

[ABSTRACT]

[Means for Solution]

5 A heat resistant coated member comprising a substrate made  
of a material selected from the group consisting of Mo, Ta, W,  
Zr, and carbon and a coating of rare earth-containing oxide thereon,  
the rare earth-containing oxide coating including a surface layer  
having a hardness of at least 50 HV in Vickers hardness.

[Effect]

10 In addition to having a good heat resistance, the coated  
member of the invention also has a good corrosion resistance and  
non-reactivity, and can therefore be effectively used for sintering  
or heat-treating metals or ceramics in a vacuum, an inert atmosphere  
or a reducing atmosphere. Where the surface layer of the rare  
15 earth-containing oxide coating has a Vickers hardness of at least  
50 HV, the rare earth-containing oxide coating is prevented from  
peeling from the substrate. Where the oxide coating has a surface  
roughness of up to 20  $\mu\text{m}$  in centerline average roughness Ra, it  
becomes effective for preventing a metal or ceramic product from  
20 deforming during sintering or heat treatment.

[Selected Drawing] none